

Microstructure and High Temperature Hoop Creep Performance of ODS Alloys

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Today's presentation themes

- Improving ODS-FeCrAl alloy hoop creep response
 - *Thermo-mechanical routes to altering the underlying grain shape*
 - *Motivation for cross-rolling and flow-forming*
- Understanding the precise role of flow forming
 - *Whether grain fibering or simply cold-working*
- Exploring Impurity content in ODS FeCrAl alloys
 - *Does chemistry, oxide content play a role?*



Experimental Hoop Creep Test Regime

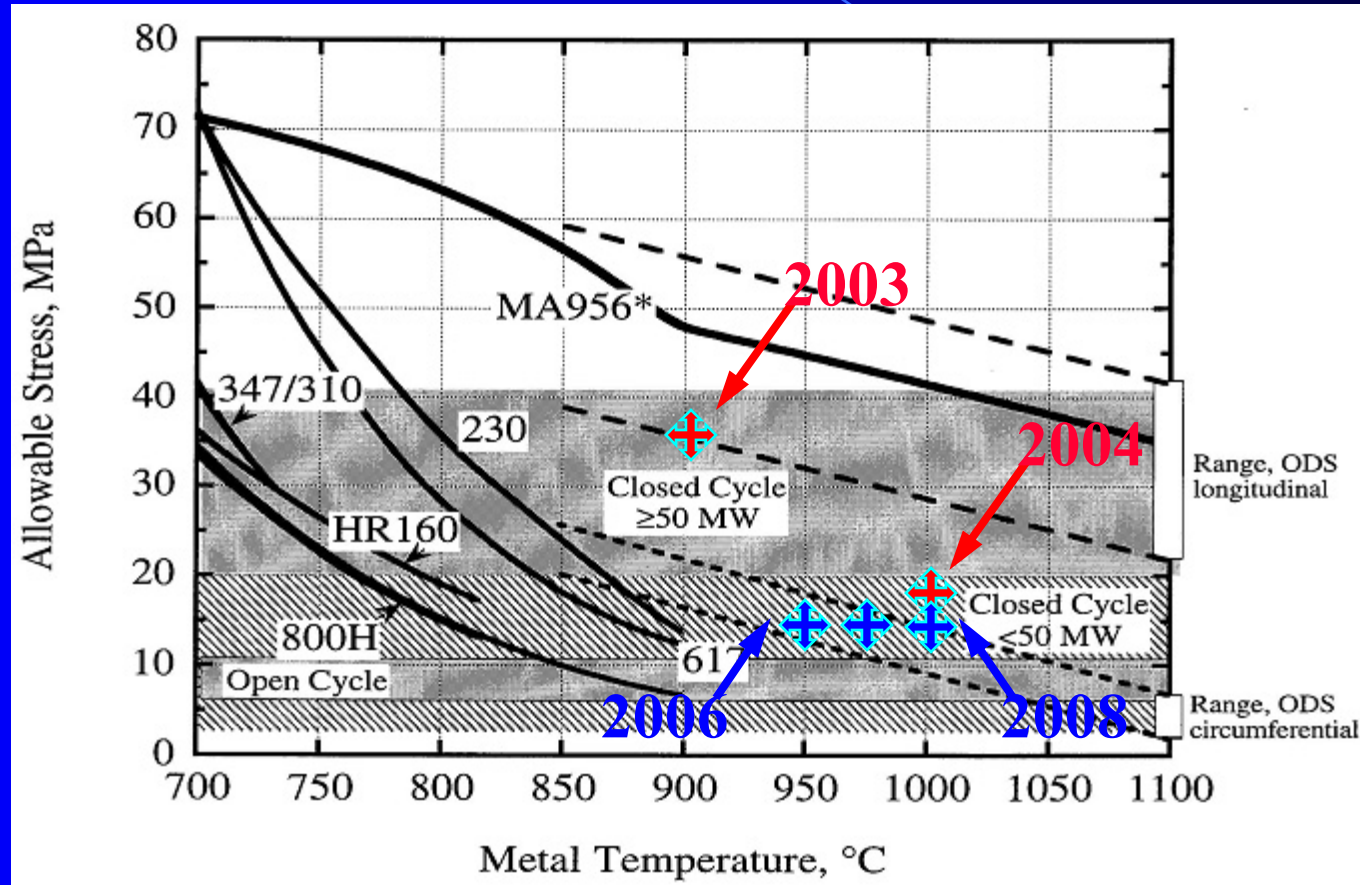
- The baseline Larsen-Miller Parameter for ODS MA956 hoop creep at 900°C, 2Ksi = 46
- Test samples cut in transverse orientation from flattened tubes
- All tests performed at 2Ksi for comparison of creep rates, creep life and failure mode
- *Status Today: increased LMP from 46 to >55*

$$LMP = 0.001(Temp^{\circ}F + 460)(20 + Log(life))$$



Hoop Creep Test Status for ODS Alloys

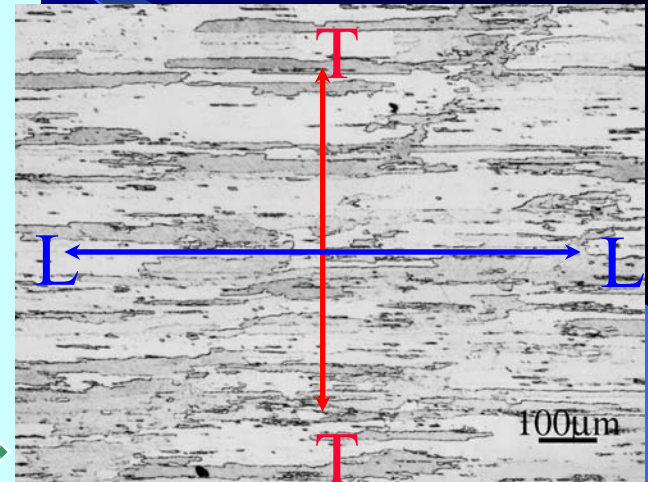
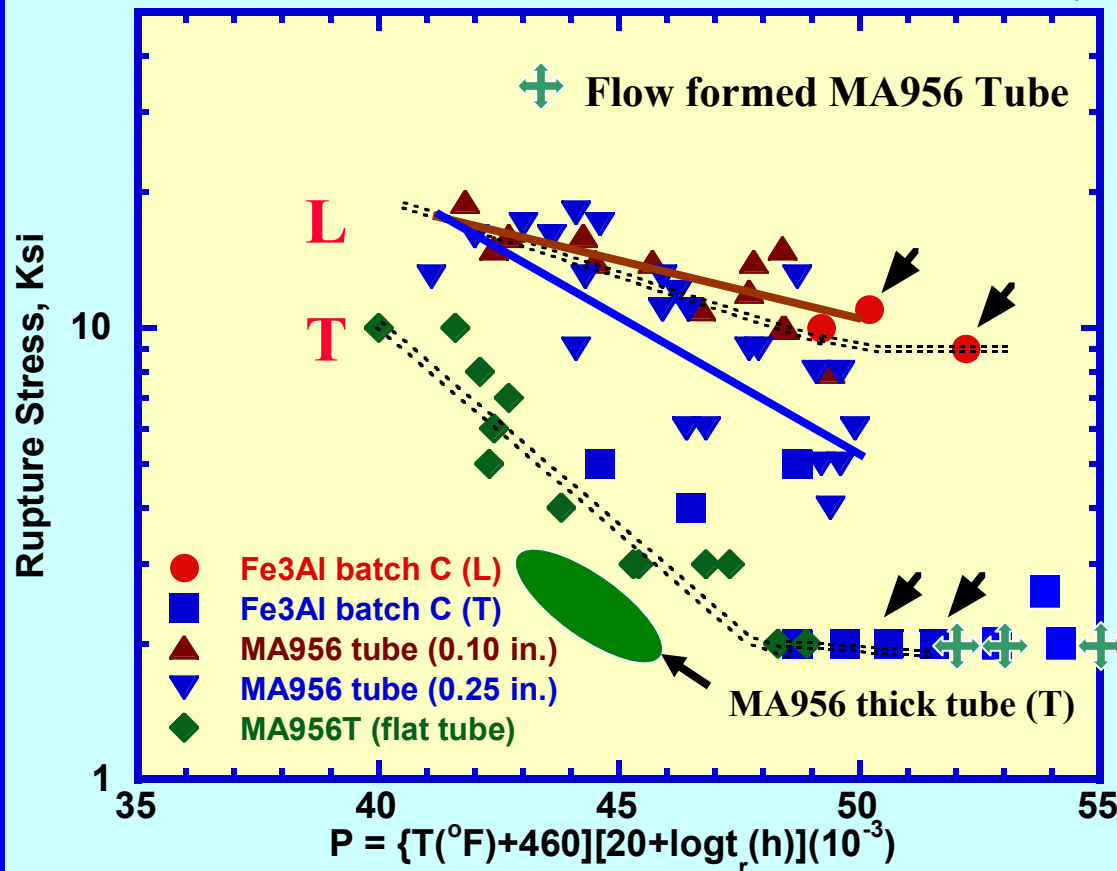
Data points only for test surviving at least 6 months on the test rig



- ✚ Current hoop creep metrics for ODS-Fe₃Al tubes
- ✚ Current hoop creep metrics for flow formed MA956 tubes

Creep Comparison for ODS-Materials

Longitudinal and Hoop Creep Anisotropy in MA956 and ODS-Fe₃Al

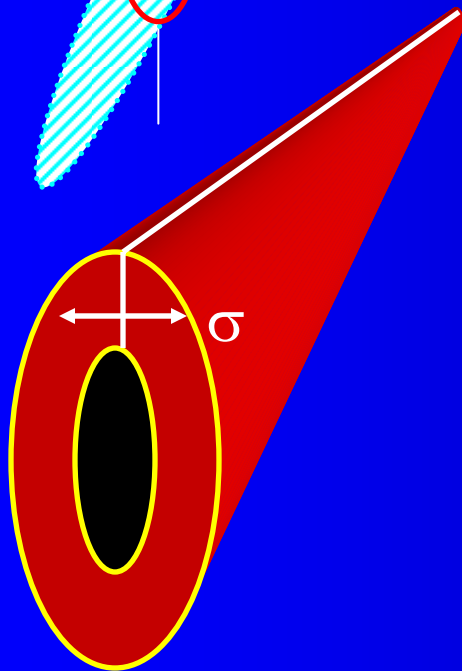
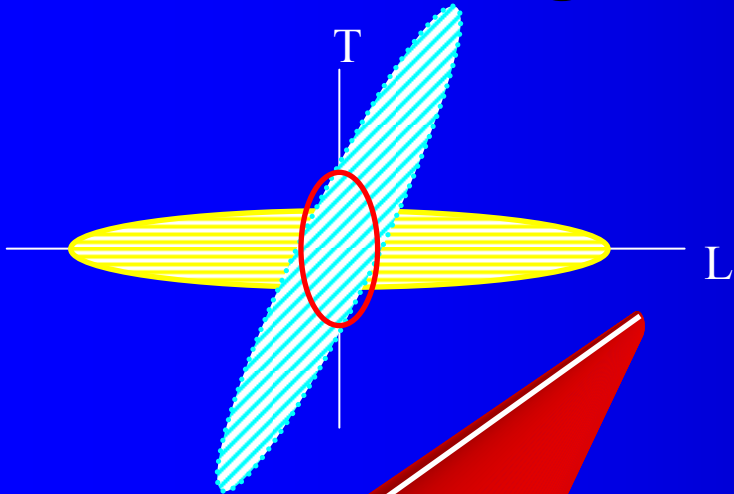


i) L superior than T

ii) ODS-Fe₃Al superior than MA956

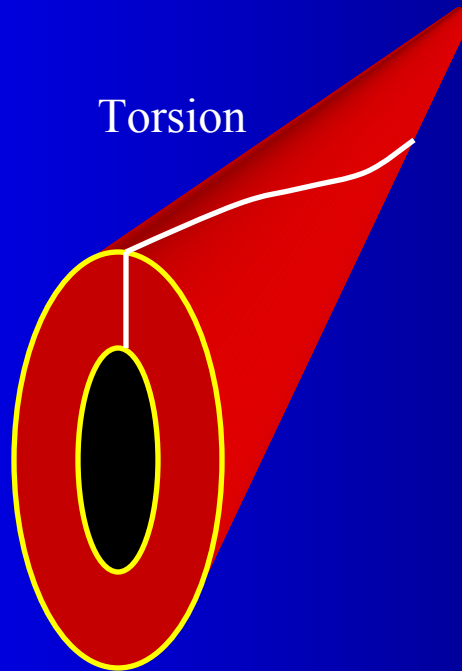
iii) Thin walled MA956 better than thick walled MA956

Grain realignment in the hoop direction



As processed grain boundaries normal to hoop loading axis

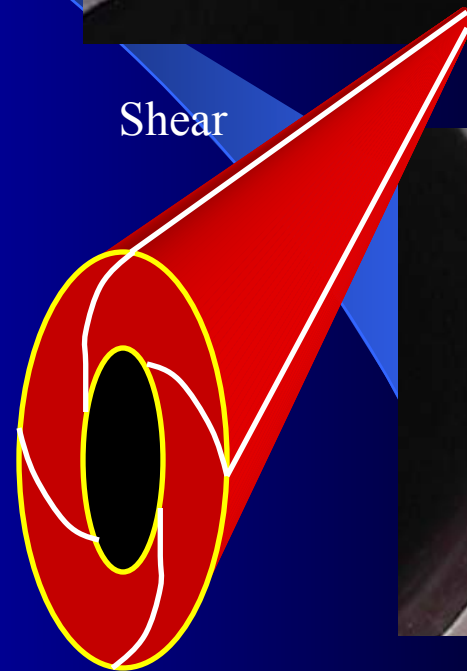
Torsion



Grain realignment along length of tube



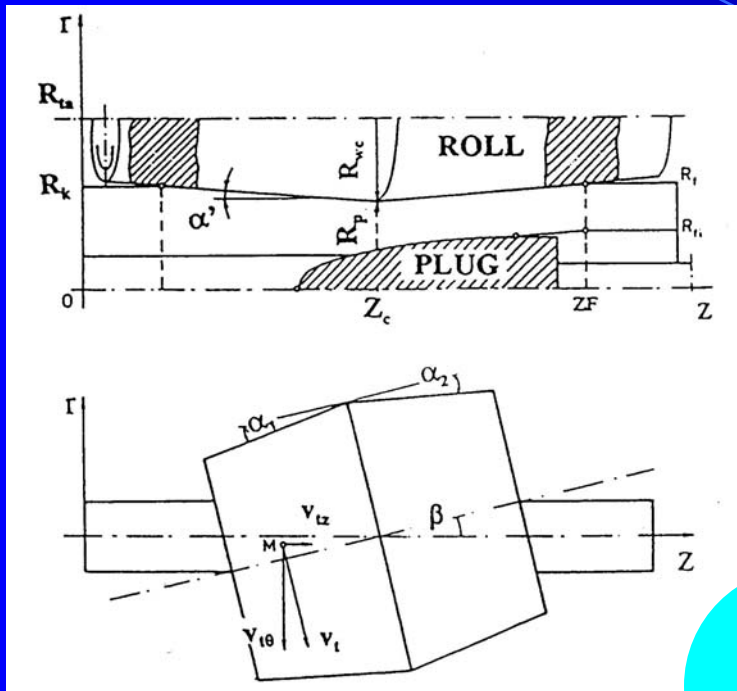
Shear



Grain realignment along radius of tube

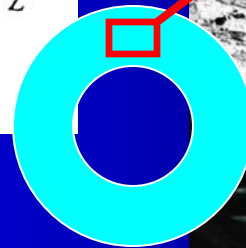
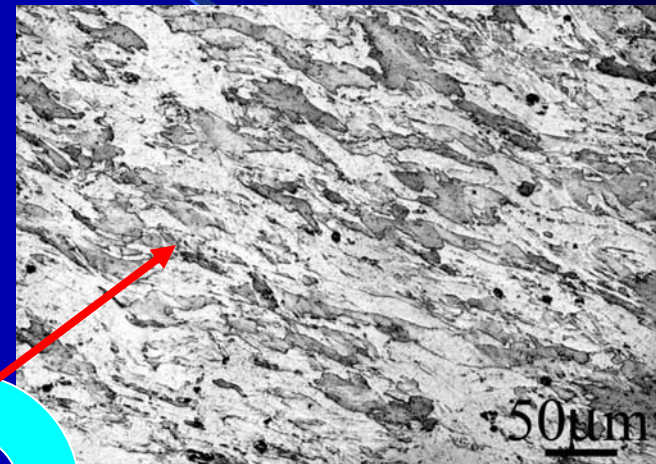


Helical rolling techniques



$$V_{tz} = V_t \sin \alpha = \omega R_{\omega Z} \sin \beta$$

$$V_{t\theta} = V_t \cos \alpha = \omega R_{\omega Z} \cos \beta$$



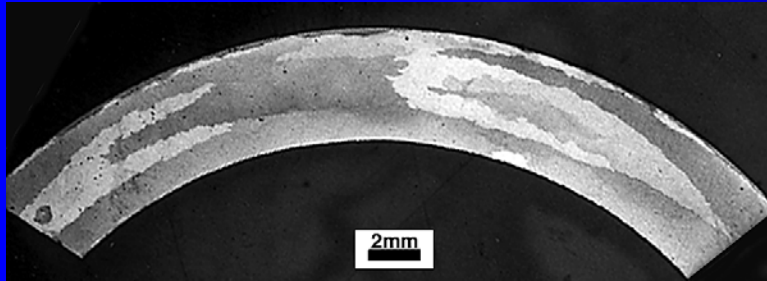
Grain shapes can be altered via torsion techniques.

Such helical grain modification is anticipated to improve hoop creep response.

Improved Grain Aspect Ratio



Desired Microstructure

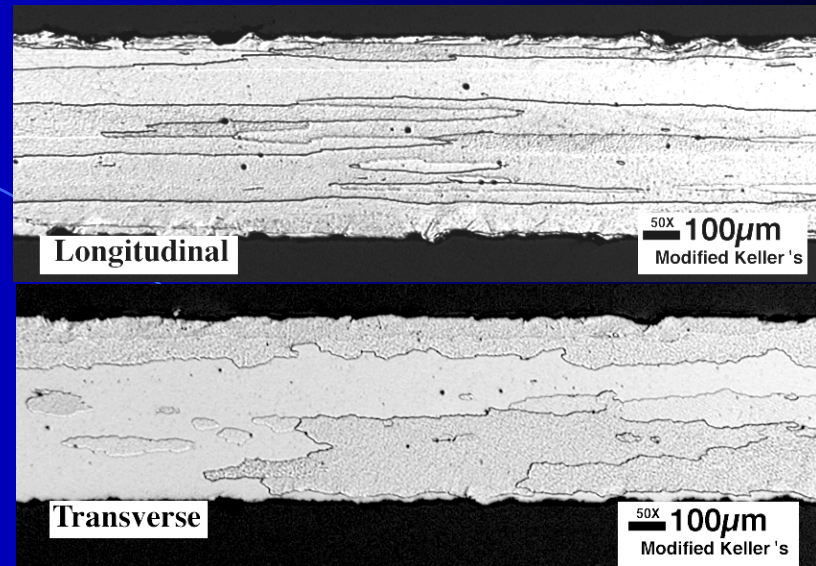


ODM-751, Onion-skin grain structure

In ODS MA956, coarse, secondary recrystallized, grain structure was only possible after extreme cold-working via flow forming.

Flow forming does NOT produce any fibering.

Only contribution is Cold Working

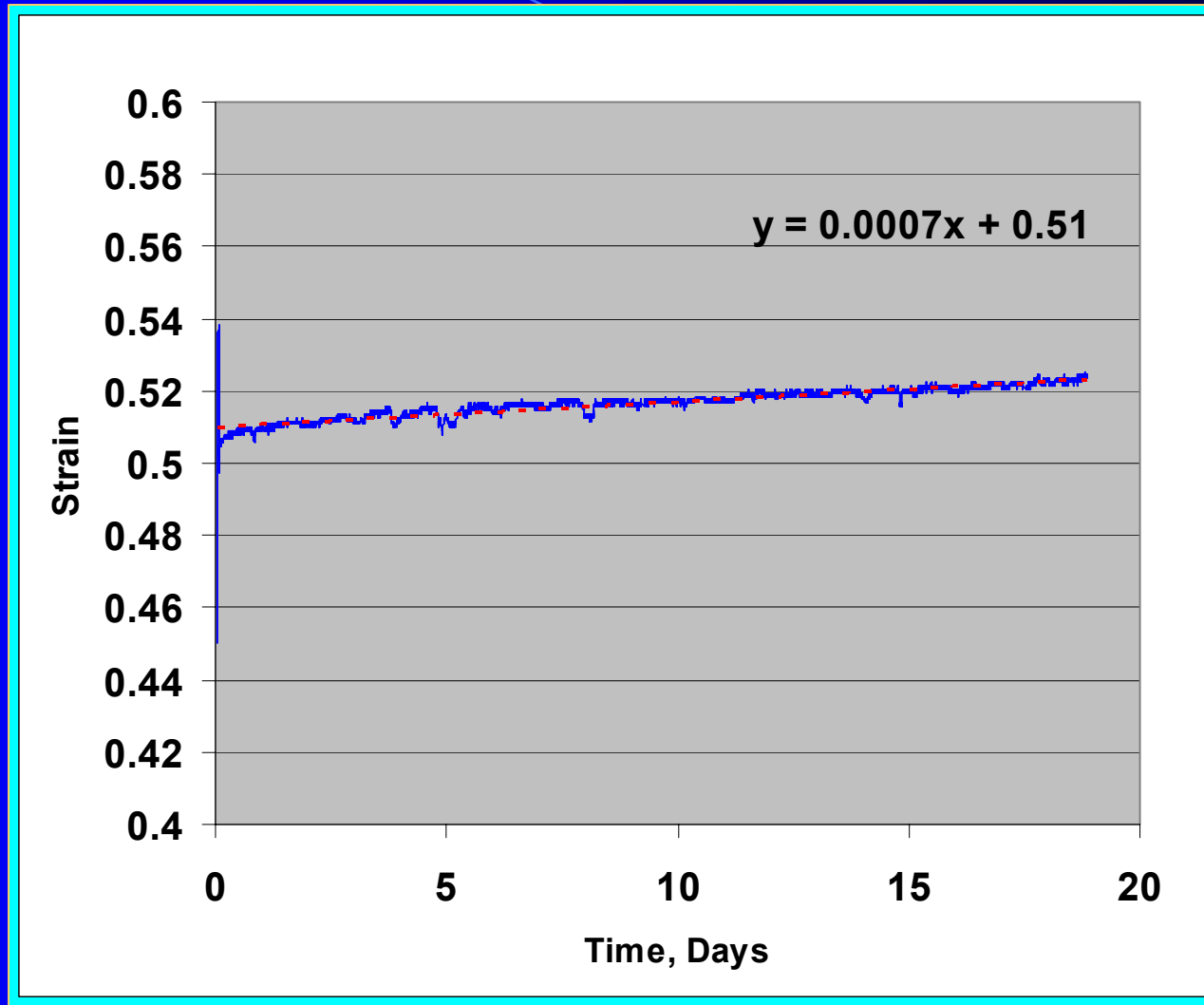


MA956, flow formed grain structure



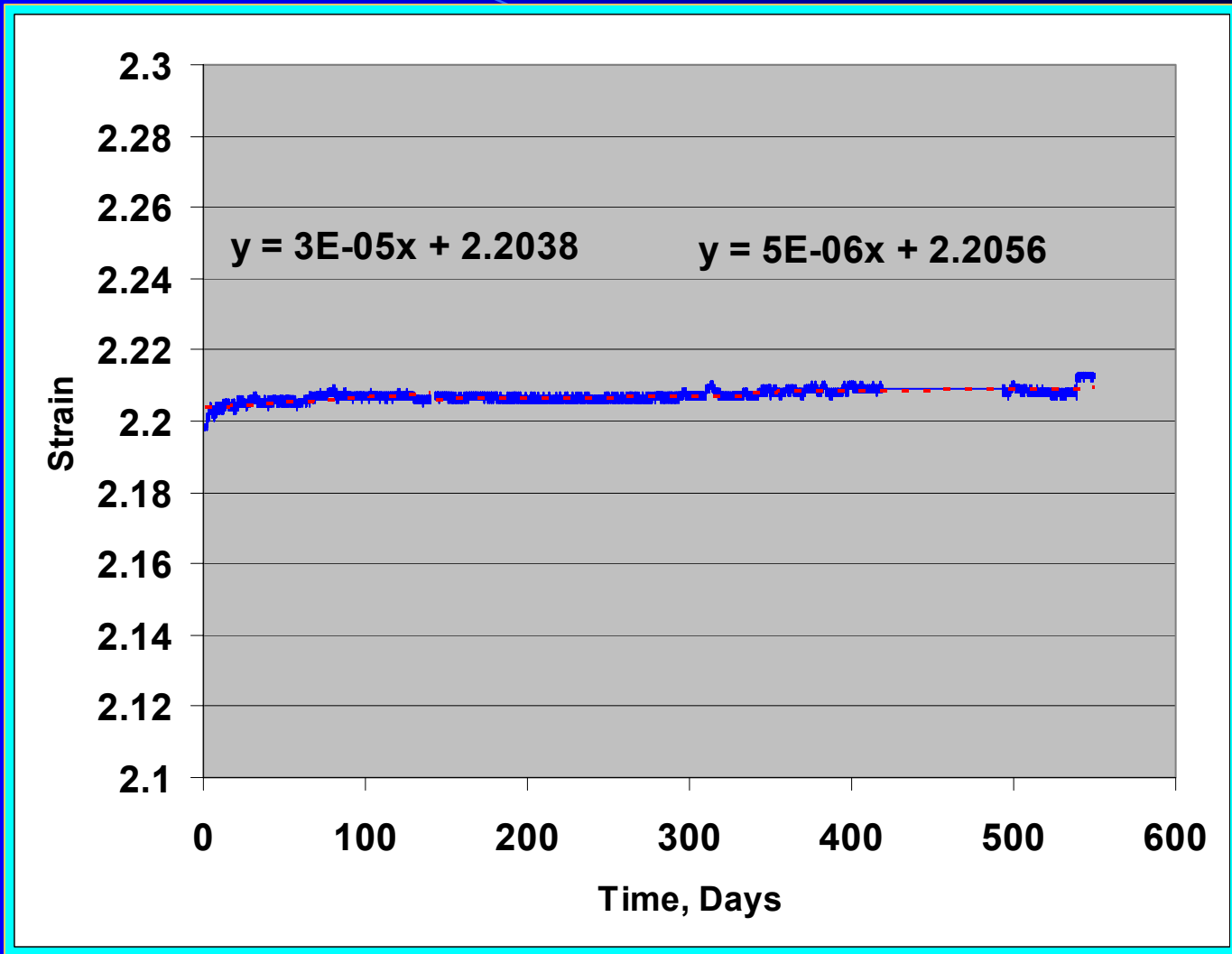
MA956, starting tube 0.25" thick wall. *flow formed* tube 0.03-0.04"

Classical MA956 Hoop Creep Curve



As-received recrystallized tube: Hoop creep test at 900°C , 2Ksi stress, in air. Observed Creep rate = 7.0×10^{-4} /day

Flow Formed MA956 Hoop Creep



**Tube Flow Formed 85% reduction in tube wall thickness:
Recrystallized at 1375°C in air. Creep test at 900°C , 2Ksi
stress, in air. Observed Creep rate range= 3.0×10^{-5} - 5.0×10^{-6} /day**



Hoop Creep: Processed MA956 Tubes

Test	MA956 Alloy Treatment & HT	Temp	Stress	Life, hrs	LM Para	rate/day
1	MA956 Tube As-Is, HT:1375°C-1hr, Air	900°C	2Ksi		46.09	2.00e ⁻²
2	MA956 Tube As-Is, HT:1375°C-1hr, Air	900°C	1Ksi		48.81	2.00e ⁻⁵
3	CR-20%@900C, HT: 1375°C-1hr, Air	900°C	2Ksi		48.87	9.00e ⁻⁵
4	CR-20%@900C, HT: 1375°C-1hr, Air	900°C	2Ksi		48.24	6.00e ⁻⁴
5	CR-20%@900C, HT: 1375°C-1hr, Air	900°C	2Ksi		48.89	1.00e ⁻⁴
6	CR@900C, $\beta=8^\circ$, HT:1375°C-1hr, Air	900°C	2Ksi		46.89	5.70e ⁻³
7	FlowForm@RT, HT:1375°C-1hr, Air2	1000°C	2Ksi	452	51.93	7.00e ⁻⁴
8	FlowForm@RT, HT:1375°C-1hr, Air2	950°C	2Ksi	7329	52.55	2.00e ⁻⁵
9	FlowForm@RT, HT:1375°C-1hr, Air2	975°C	2Ksi	20922*	54.65	5.00e ⁻⁶
10	FlowForm@RT, HT:1375°C-1hr, Air2	1000°C	2Ksi	16757	55.52	7.00e ⁻⁶

* Data Collection continuing on Test 9.



Why do ODS-MA956 need much help?

***Coarse grain sized material is quite OK
but rather expensive path to get there***

- Extremely large strains are required.
- Cold work seems to produce better result than hot working & grain fibering
- Better bang from large grain size alone.

explore alternate ODS-MA956 chemistry?

- In parallel universe of ODS-Fe₃Al large grain structure are possible – without any special, or expensive treatments.



Impurity Issues in powder milling processes:

*Lessons: milling derived impurity in ODS-Fe₃Al program:
elevated impurity renders materials of subpar properties*

Element	As received		milled powder batches		
	HM	PM	PMWY-1	PMWY-2	PMWY-3
Fe	Bal.	79.6			
Al	16.3	18.20			
Cr	2.4	2.18			
Zr	20 ppm	26 ppm			
O (total)	60 ppm	110 ppm	1800 ppm	1900 ppm	1400 ppm
O (in Y ₂ O ₃)			1025 ppm	1053 ppm	1080 ppm
O balance			775 ppm	847 ppm	320 ppm
O pickup			665 ppm	737 ppm	210 ppm
N	18 ppm	7 ppm	1264 ppm	145 ppm	88 ppm
N pickup			1257 ppm	138 ppm	81 ppm
C		24 ppm	667 ppm	360 ppm	303 ppm
C pickup			643 ppm	336 ppm	279 ppm
H		16 ppm	115 ppm	40 ppm	29 ppm
C+N+O pickup			2565 ppm	1211 ppm	570 ppm

Bulk compositions are identified in wt%, HM and PM are two separate analyses of atomized Fe₃Al powder

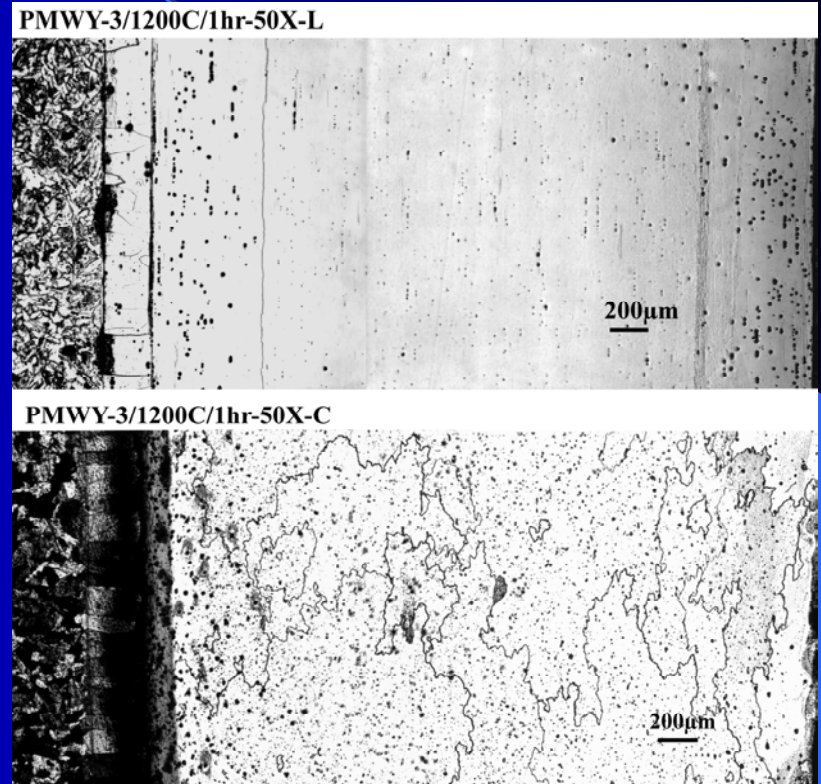
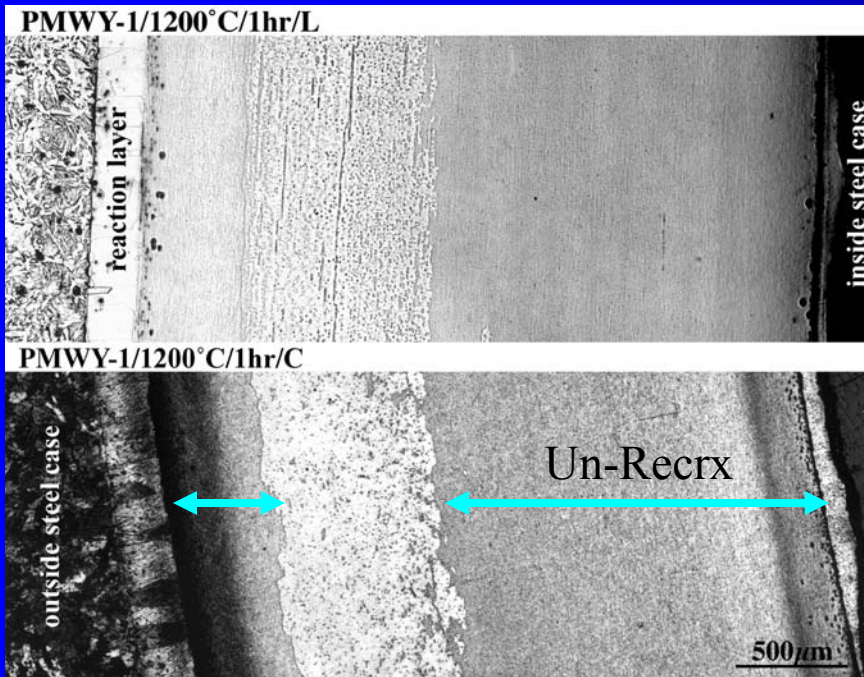


Parallel Universe of ODS-Fe₃Al Alloys

Oxide content, impurities affect recrystallization

High impurity powder batch: PMWY1

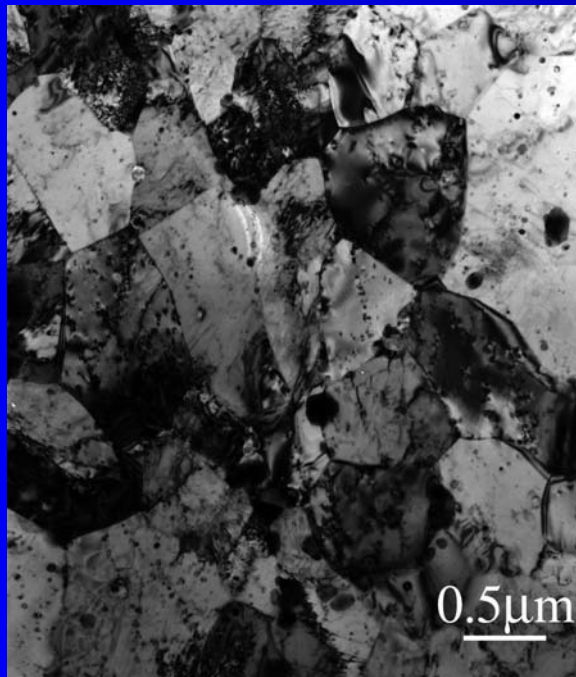
Low impurity powder batch: PMWY3



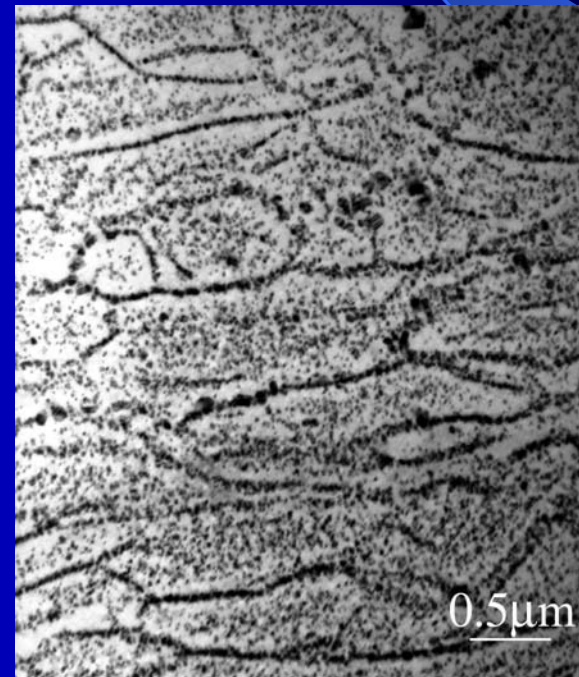
Powder batches with high interstitial impurity are particularly resistant to static recrystallization treatments. Efforts to increase time-temperature combinations have met with only marginal success.

Parallel Universe of ODS-Fe₃Al Alloy

Large recrystallized grain size achieved via control of IMPURITY & OXIDE CONTENT without requiring any additional deformation. High oxide content produced small pinned grain similar to that seen in ODS-FeCrAl alloys



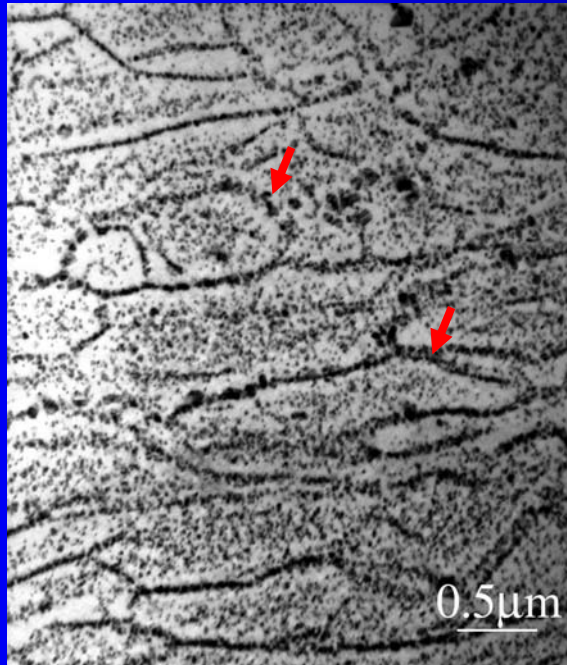
High oxide, impurity
content; Small Grains



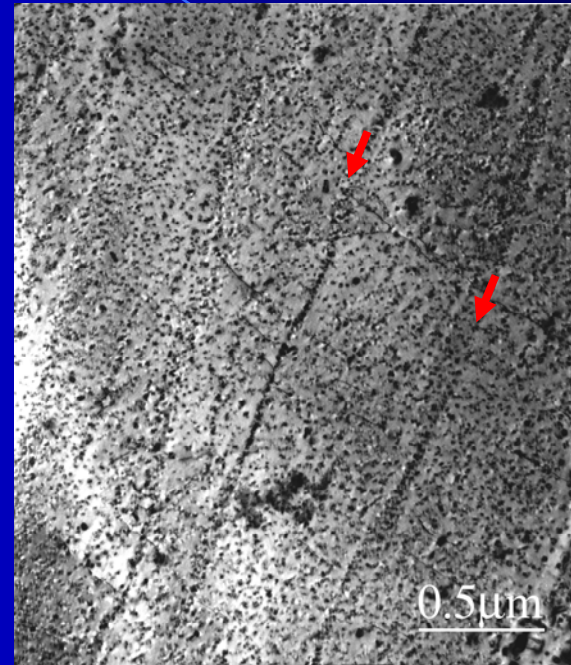
Low oxide, impurity
content; Large Grains

Recrystallization Microstructures

Transverse view

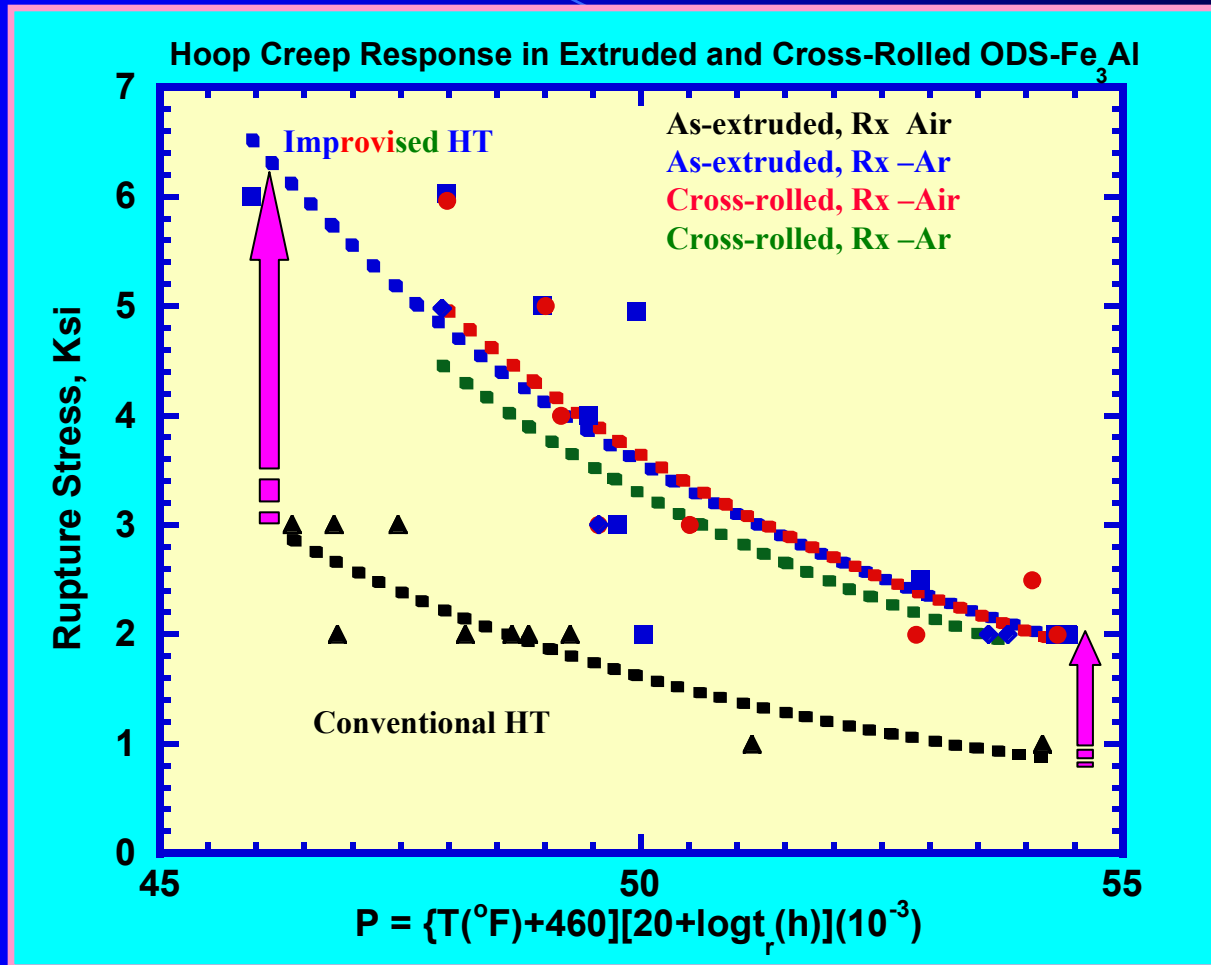


Longitudinal view



Specimens extracted from the high purity powder chemistry (PMWY-3) heat-treated tubes (HT: 1200°C-1hr in Air). **Red Arrows** indicate prior particle boundaries in transverse and longitudinal views

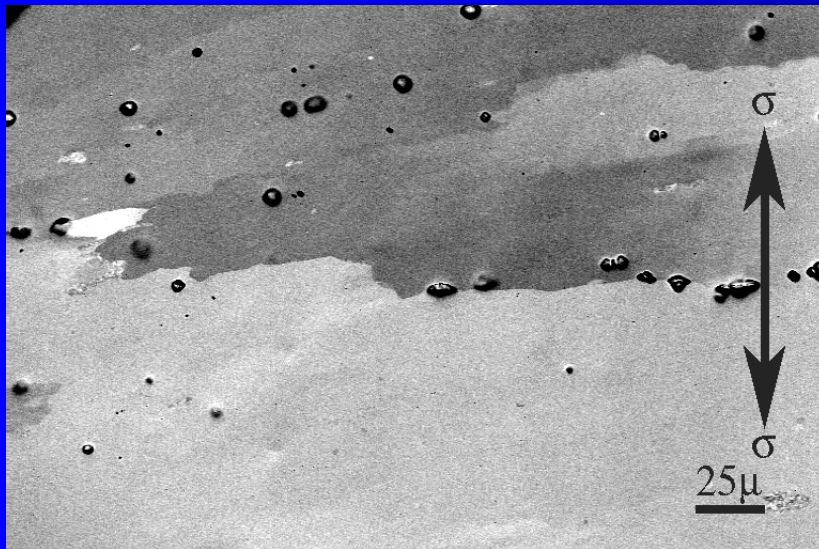
Improvements in ODS-Fe₃Al Hoop Creep



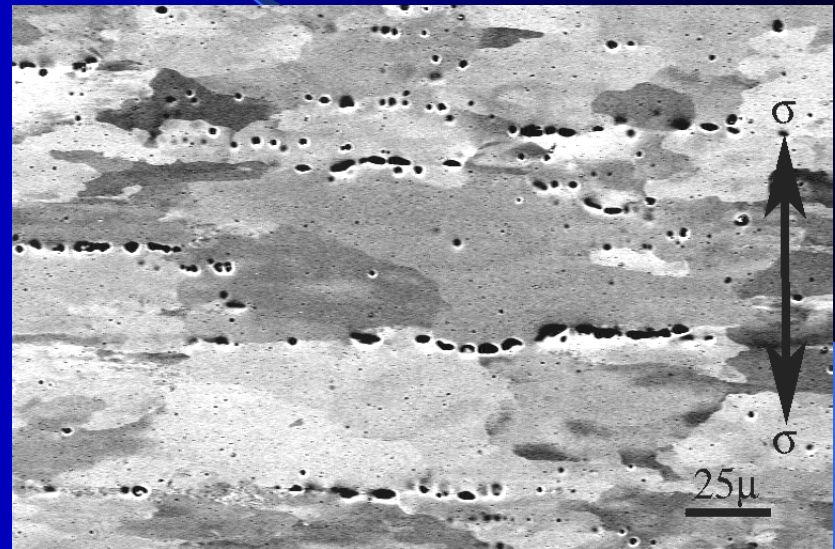
Hoop creep response is improved by control of recrystallization environment and temperature (low cost). Tests of cross rolled materials show significant improvements as well (high cost).

Transverse Creep: Failure Microstructures

The dirt on ODS alloys



PMWY-3, transverse creep @ 1000°C

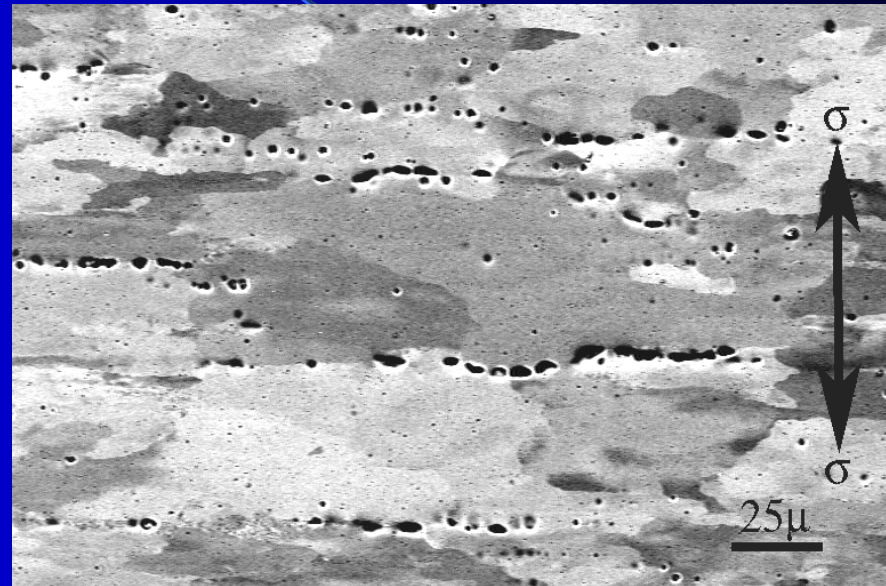
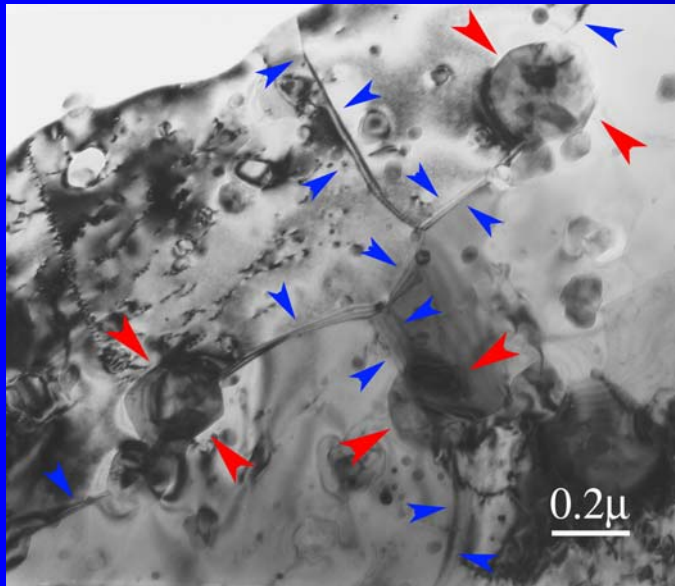


MA-956, transverse creep @ 900°C

Low impurity oxides in PMWY-3 produce large recrystallized grains which respond better in transverse creep testing. Creep void formation is suggested to occur in the vicinity of large impurity oxide particles and/or stringers.

Transverse Creep: Failure Microstructures

The dirt on ODS alloys

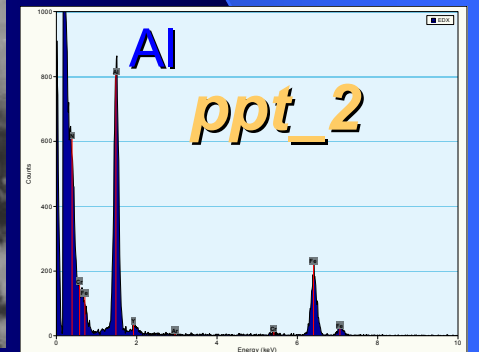
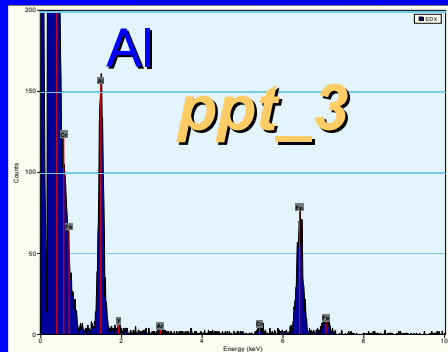
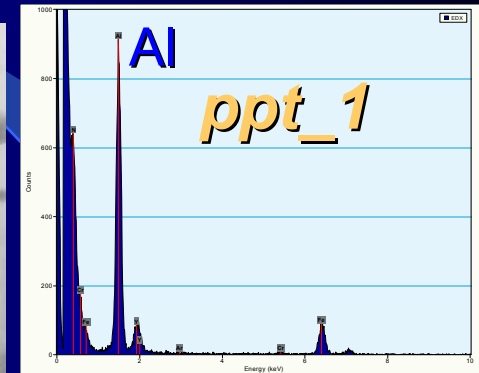
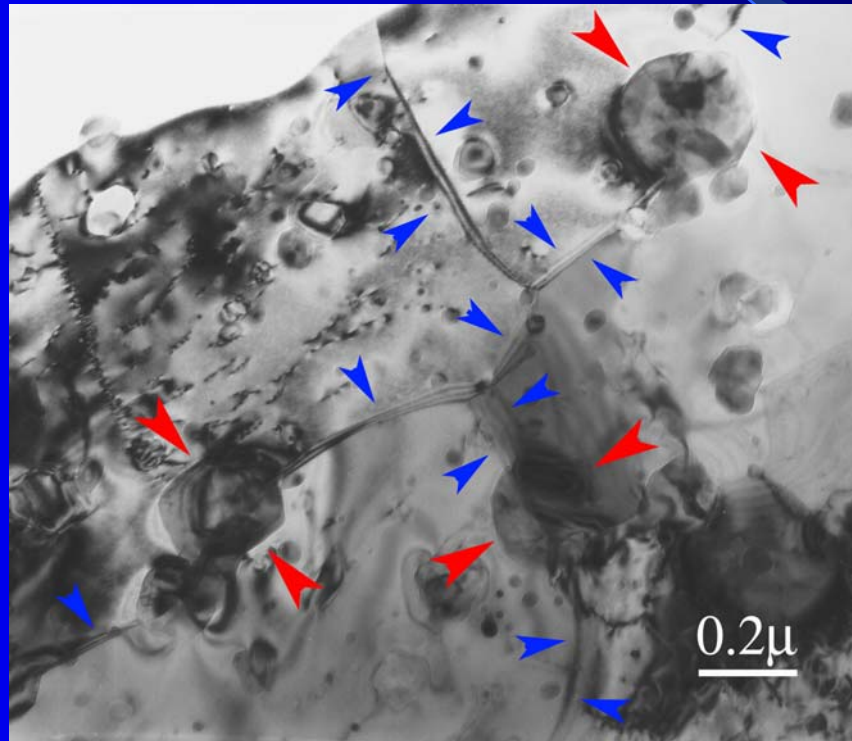
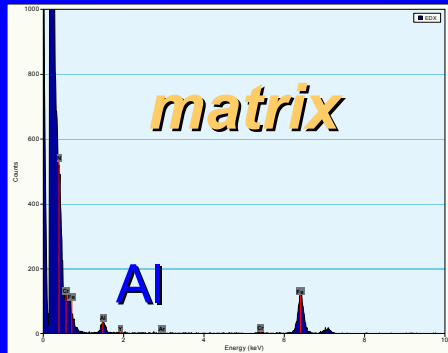


MA-956, transverse creep @ 900°C

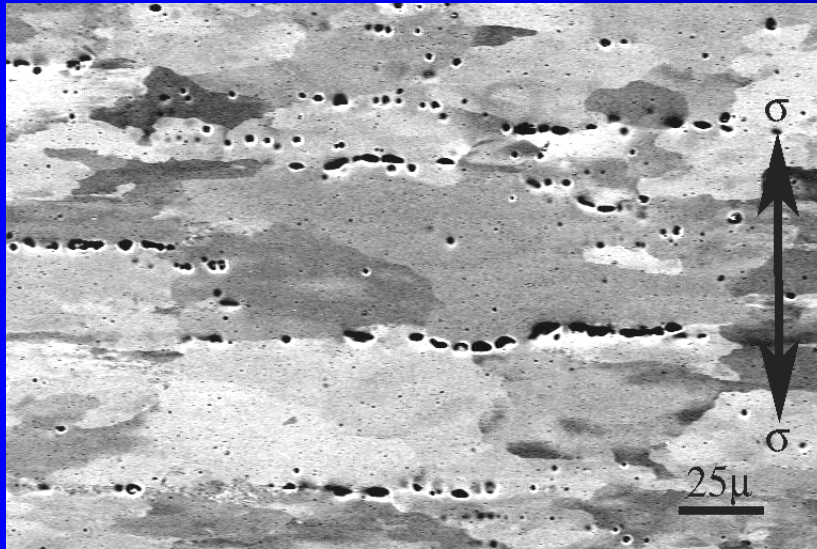
Creep void formation is suggested to occur in the vicinity of large impurity oxide particles and/or stringers. Low impurity oxides in ODS-Fe₃Al produce large re-crystallized grains which respond better in transverse creep testing.

MA956 has larger vol. frac. of yttria precipitates than ODS-Fe₃Al Alloy

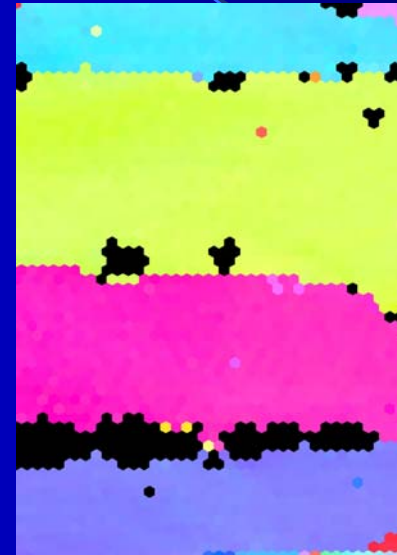
Restricted Grain Growth: Grain Boundary Pinning by Impurity Al-Oxides, Al-Nitrides



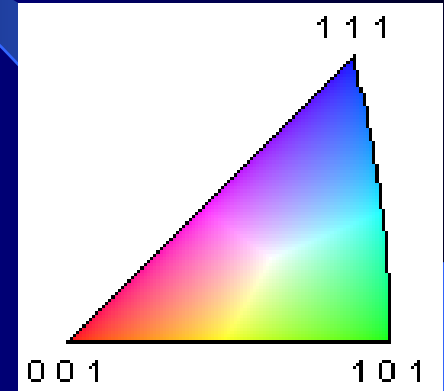
EBSD Mapping of Transverse Creep: GB Creep Voids as $fn(\text{Grain Misorientation})$



MA-956, transverse creep @ 900°C



EBSD, Tube Axis



Orientation Legend

High angle boundaries about the tube axis are susceptible to increased grain boundary void formation and coalescence. Creep void formation is suggested to occur in the vicinity of large impurity oxide particles and/or stringers.

MA956 has larger vol. frac. of yttria precipitates than ODS-Fe₃Al Alloy

Chemistry of Available Ferritic ODS Alloys

Alloy	Density, gm/cm ³	Fe	Cr	Al	Ti	Si	RE
SMC MA-956		Bal	20.0	4.5	0.5	-	0.5, Y ₂ O ₃
SMC MA-956HT	7.20	Bal	21.6	5.9	0.4	0.07	0.5, Y ₂ O ₃
PM 2000	7.18	Bal	20.0	5.5	0.5	-	0.5, Y ₂ O ₃
ODS-Fe ₃ Al	6.53	Bal	2.2	15.9	0.07	0.1	0.5, Y ₂ O ₃

Yttria addition is made by wt%. In high chromium FeCrAl denser alloy this has the net effect of increasing dispersion vol. frac. (f) by 10-15%. This affects the pinned grain size and may dictate secondary recrystallization.

Zener-Smith eq. for predicting grain size controlled by particle pinning:

$$D_{max} \leq 4r/3f \quad D = \text{grain size}, f = \text{ppt. vol. frac}$$



Summary of Results

- A firm threshold established for ODS-FeCrAl (MA956) transverse creep tested at 2Ksi at 950-1000°C.
- Grain fibering, flow-forming methods offer significant hoop creep enhancement
- Flow forming contributes to cold-work only and produces little to no fibering in the hoop direction.
- ODS MA956 resistance to secondary recrystallization may stem from impurity chemistry – primarily from milling. Our in house example of milling studies in ODS-Fe₃Al indicates as such– but similar studies for commercial ODS FeCrAl alloys still not available.

